भारतीय मानक
Indian Standard

IS 5551 : 2023

वारफ़रीन-सोडीयम, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Warfarin-Sodium, Technical — Specification

(First Revision)

ICS 65.100.99

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

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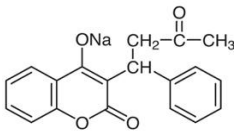
FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

The sodium salt of 3-acetonylbenzyl-4-hydroxycoumarin, namely, 'Warfarin-Sodium' is used in the preparation of formulations for the control of rodent pests, such as rats, mice and bandicoots. The material acts as an haemorrhagic agent or as an anti-coagulant, which when eaten for a period of time, causes death due to haemorrhage in the blood system.

The structural and chemical formulae and the molecular weight of the compound are given below:

Empirical Formula	*Structural Formula*	*Molecular Weight*
$C_{19}H_{15}O_4Na$		333.0

Warfarin-sodium, technical is known to have two impurities, namely, Alice's ketone [3-(O-hydroxyphenyl)-5-phenyl-2-cyclohexene-1-one] and benzalacetone, alkali insolubles. These impurities, when present beyond a certain limit, tend to repel the rats from accepting the bait containing this product. On the basis of the results of investigations, this standard prescribes the maximum limit of Alice's ketone as 750 ppm in warfarin-sodium, technical.

The standard was first published in 1970. In this revision, the standard has been brought out in the latest style and format of the Indian Standards, and references to Indian Standards wherever applicable have been updated.

This standard contains clauses **4.1**, **F-2.3** and **F-3.4** which call for an agreement between the purchaser and vendor.

In the preparation of this standard due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

The composition of the Committee responsible for the formulation of this standard is listed in Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

WARFARIN-SODIUM, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of test for warfarin-sodium (sodium salt of 3-acetonylbenzyl-4-hydroxycoumarin), technical.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves (*fourth revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 10946 : 1996	Methods of sampling for technical grade pesticides

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a free-flowing, yellow to brownish, crystalline powder, free from any lumps, extraneous impurities, added modifying agents or odours which may limit its suitability for baiting rodents or impair its effectiveness.

3.2 The material shall comply with the requirements specified in Table 1.

4 PACKING

The material shall be packed in clean and dry air-tight containers made of galvanized steel sheet, tinplate, steel, glass or plastic as agreed to between the purchaser and the manufacturer.

5 MARKING

5.1 The containers shall be securely closed and shall be bear legibly and indelibly the following information:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Active ingredient content, percent (*m/m*);
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology (Packaged Commodities) Rules*, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

Table 1 Requirements for Warfarin-Sodium, Technical (*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Refer to Annex
(1)	(2)	(3)	(4)
i)	Sodium salt of 3-acetonylbenzy1-4-hydroxycoumarin, percent by mass, *Min*	92.0	A
ii)	Sieving requirement, material passing through 151-micron IS Sieve, percent by mass, *Min*	95.0	B
iii)	Moisture content, percent by mass, *Max*	1.0	C
iv)	Material insoluble in distilled water at 30 ºC, percent by mass, *Max*	0.2	D
v)	Alice's ketone, ppm, *Max*	750	E

6 SAMPLING

Representative samples of the material shall be drawn according to IS 10946.

7 TESTS

7.1 Tests shall be carried out as prescribed in the appropriate appendices specified in co1 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A
[*Table* 1, *Sl No.* (i)]

DETERMINATION OF SODIUM SALT OF 3-ACETONYL-BENZYL-4-HYDROXYCOUMARIN

A-1 APPARATUS

A-1.1 Spectrophotometer — Ultra-violet, with 1 cm quartz cells.

A-2 REAGENT

A-2.1 Standard Sodium Hydroxide Solution — 0.1 N.

A-3 PROCEDURE

Weigh accurately about 50 mg of the sample, transfer the same to a 100 ml volumetric flask, add standard sodium hydroxide solution to effect solution, and make up to the mark with the same. Dilute 2 ml of this solution to 100 ml with the standard sodium hydroxide solution. Set the spectrophotometer at maximum sensitivity and determine the absorption of the final solution at 308 nm using standard sodium hydroxide solution as reference.

A-4 CALCULATION

Molecular weight of sodium salt of warfarin = 330.3

Molar extinction coefficient (*see* Note) = 1.42 X 10^4

Sodium salt of 3-acetonylbenzyl-4-hydroxycoumarin content, percent by mass

$$= \frac{E \times 330.3 \times 100 \times 100 \times 100}{1.42 \times 10\,000 \times 2 \times W} = \frac{E \times 11.6}{10}$$

where

E = absorption of the final solution at 308 nm, and

W = weight of sample (g)

NOTE — The molar extinction coefficient should be determined with pure sodium salt of 3-acetonylbenzyl-4-hydroxycoumarin. The value given here is an example only.

ANNEX B
[*Table* 1, *Sl No.* (ii)]

TESTING FOR SEIVING REQUIREMENT

B-1 APPARATUS

B-1.1 Test Sieve — 151 micron IS Sieve [*see* IS 460 (Part 1)], prepared for test by removing any film, grease or other water-repellent material and then drying.

B-1.2 Weighing Dish — tared, one.

B-2 METHOD

Weigh accurately 100 g of the material and transfer it to the test sieve. Cover the sieve and screen the material in a suitable sieve vibrator for 10 minutes. Two small square rubber cubes are introduced along with the material on the sieve to facilitate the breaking up of any soft lumps of the caked material.

After 10 minutes stop the machine and brush the residue on the sieve into a tared weighing dish. Weigh the dish and determine the weight of the residue.

B-3 CALCULATION

Material passing through l5l micron IS Sieve, percent by mass $= \frac{100\ (1-w)}{W}$

where

w = weight in g of the material retained on the test sieve, and

W = weight in g of the material taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF MOISTURE CONTENT

C-1 PROCEDURE

Weigh accurately 2 g of the sample into a dry tared dish of nickel, platinum or aluminium. Keep the dish in an air-oven maintained at (101 ± 1) °C for 10 hours. Afterwards, cool in a desiccator and weigh. Dry the material again for one hour. Cool and repeat this process until the change in weight is not greater than 2 mg. Report the loss in weight as moisture content (*see* **C-2**).

C-2 CALCULATION

$$\text{Moisture content, percent by mass} = \frac{100(W-w)}{W}$$

where

W = mass in g of the material taken for the test, and

w = mass in g of the material after drying

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF MATERIAL INSOLUBLE IN DISTILLED WATER

D-1 PROCEDURE

Weigh accurately 5 g of the sample into a 250 ml beaker, add 50 ml of distilled water and heat it to 30 °C while stirring until all the soluble material is dissolved. Filter the solution through a dry tared Gooch or sintered glass crucible of porosity No. 3 and wash with 100 ml portions of distilled water at approximately 30 °C. Dry to a constant weight in an oven at 110 °C. Cool and weigh.

D-2 CALCULATION

$$\text{Material insoluble in distilled water at 30 °C, percent by mass} = \frac{100\times w}{W}$$

where

w = weight in g of the residue after drying, and

W = weight in g of the material taken for the test.

ANNEX E

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF ALICE'S KETONE IN WARFARIN-SODIUM, TECHNICAL

E-1 PROCEDURE

Dissolve 1.17 g of sample of warfarin-sodium, technical in 10 ml of 5 percent aqueous sodium hydroxide solution. Determine the optical density in a Beckman DU spectrophotometer (or similar instrument) at 385 nm through a 1 cm light path. The parts per million of 'Alice's ketone' is 380 x optical density.

ANNEX F
(*Foreword*)

COMMITTEE COMPOSITION
Pesticides Sectional Committee, FAD 01

Organization	*Representative(s)*
Directorate of Plant Protection Quarantine and Storage, Faridabad	DR RAVI PRAKASH (***Chairperson***)
All India Biotech Association, New Delhi	SHRI SAURABH SINGHAL SHRI SHAH JI DHAR (*Alternate*)
Central Insecticide Board and Registration Committee, Faridabad	SECRETARY DR VANDANA SETH (*Alternate*)
Central Insecticide Laboratory, Faridabad	DR ARCHANA SINHA SHRI SUBHASH CHAUDHARY (*Alternate*)
Consumer Guidance Society of India, Mumbai	SHRI SITARAM DIXIT DR M. S. KAMATH (*Alternate*)
Crop Care Federation of India, New Delhi	DR J. C. MAJUMDAR
Crop Life India, New Delhi	SHRI ASITAVA SEN MS NIRUPAMA SHARMA (*Alternate*)
CSIR-Indian Institute of Toxicology Research, Lucknow	DIRECTOR DR SHEELENDRA P. SINGH
Food Safety and Standards Authority of India, New Delhi	ADVISOR (STANDARDS)
FMC India Private Limited, Bengaluru	SHRI CHIRAG PATEL
IDMA Laboratories Limited, Chandigarh	DR INDRA RAI
Indian Agricultural Research Institute, New Delhi	DIRECTOR
Indian Institute of Packaging, Mumbai	DR TANWEER ALAM
Indian Pest Control Association, New Delhi	SHRI UDAYAN GHOSH
Institute of Pesticide Formulation Technology, Gurgaon	DR M. VAIRAMANI
Ministry of Agriculture, Department of Agriculture, Chennai	JOINT DIRECTOR OF AGRICULTURE (RES) DEPUTY DIRECTOR LAB (*Alternate*)
National Centre for Integrated Pest Management, New Delhi	DR SUMITRA ARORA
National Institute of Plant Health Management, Hyderabad	DR MAHESH SAINI KUMARI T. SRIDEVI (*Alternate*)
Pesticide Manufactures and Formulators Association of India (PMFAI), Mumbai	DR ARCHANA SRIVASTAVA DR UDAY KUMAR (*Alternate*)
Regional Pesticides Testing Laboratory, Chandigarh	SHRI V. VASU

Organization	*Representative(s)*
In Personal Capacity (*4-6-90/2/8/2, Sri Devi Nilayam Tejaswinagar Coloni, Attapur, Hyderabad – 500048*)	SHRI C. V. RAO
In Personal Capacity (*263, Sector 28, Faridabad – 121008*)	SHRI VIPIN SAINI
BIS Directorate General	SHRIMATI SUNEETI TOTEJA, SCIENTIST 'E'/ DIRECTOR AND HEAD (FOOD AND AGRICULTURE) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI KULDEEP MITTAL
SCIENTIST 'B'/ASSISTANT DIRECTOR
(FOOD AND AGRICULTURE), BIS

Bureau of Indian Standards

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Review of Indian Standards

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Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	2821 8093

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